Laboratory Experiment Proposal Submission

Experimental Details

Experiment location: A127
Experiment title: Synthesis of moisture sensitive beta-NaMnO2
Experiment date: 07/06/2012
Experiment contactname: Ioanna Bakaimi, Efrain Rodriguez, Mark Green
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Chemicals Used

Chemical Name	Health	Flammability	Reactivity	Special Hazards
Na2CO3	2	0	1	BIO
Mn2O3	2	0	0	

Reactants and Resulting Samples

Chemical Name	Hazardous?	Known Hazards
NaMnO2 -pellet	N	

Required Safety Equipment

- [x] Glove Hot
- [x] Lab Coat
- [x] Tongs

Required Laboratory Equipment

- [x] Balance
- [x] Glovebox
- [x] Xray Diffractometer
- [x] Laboratory Press

Experimental Write Up

I will have displayed an A4 paper where it will be written that
"The reaction at the single zone furnace is a calcination at 950 C under continuous O2 flow. Due to the possible risks of flammability of O2 please be careful when you enter in the lab. In case you see something unusual please contact the
Control Room: 6292 or
Emergency : 2222
Ioanna Bakaimi: 8712, 00447954342930
Mark Green: 4297.
Thank you"

Procedure of the experiment:
$Na_2CO_3 + Mn_2O_3 \rightarrow 2\ NaMnO_2 + CO_2$
1) Weight appropriate amount of starting materials.
2) Grind them in the mortar and pestle for 30 min, and press powder mixture into two 20 mm diameter pellets.
3) Transfer pellets onto alumina boat and place the boat in the single zone furnace, centered.
4) Attach the appropriate SS adaptors to the alumina tube, and connect to an oxygen cylinder via rubber hoses and oxygen regulator. Attach the gas meters (pressure meters) on the O2 bottle to check the O2 pressure. It has to be around 2-3(just a little bit above zero) Attach the outlet adaptor to a bubbler filled with mineral oil to monitor gas flow and attach bubbler to exhaust located above the single zone furnace.
5) Open the oxygen tank and verify that O2 is flowing through the bubbler.
Slow the flow rate to approximately 1 bubble per 2 to 5 seconds.
6) After purging the tube with pure oxygen for approximately 30 minutes, the furnace program will be started.
7) Furnace program: heat up to 950 C with a rate of 0.4C/min, and dwell at 950 for 24hrs. During ramp rate, bubbler will be continuously monitored to ensure that the flow rate remains within the range set at roomtemperature. Upon heating, flow rate could increase or decrease, so regulator will be adjusted accordingly.
8) After 24 hours at 950 C, the sample will be quenched to room Temperature. Close the O2 bottle, so that no O2 will be flowing in the tube. Wait around 15 min to ensure that there is no O2 flow in the tube of the furnace. Then remove the adaptor, and pull the boat to the cool zone of the tube with tongs or appropriate tool.
9) 15 minutes after the quench, the sample will be removed from the furnace, placed in the dry box and ground with mortar and pestle.
10) The ground powder will then be re-pelletized, and placed back on the same alumina boat and back into the single zone furnace for the sintering step.
11) Oxygen flow will be restarted and the flow rate set to approximately 1 bubble per 2-5 seconds. After purging for 30 minutes, the furnace program will be restarted.
12) Second furnace program: ramp rate of 5 C/min to 950 C, with a dwell time of 24 hours. As in previous step, flow rate will be monitored and controlled via the oxygen regulator and bubbler.
13) Sample will again be quenched as described previously and then placed in the dry box to be ground and stored until neutron experiments.

Experimenter Signature: _____ Date: _____

Lab Responsible Signature: _____ Date: _____